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(54) INK-JET RECORDING FLUID

(57)Abstract:

PROBLEM TO BE SOLVED: To obtain an ink-jet recording fluid which solves the problem in the conventional process and gives a transparent clear image by using specified ingredients in a recording fluid comprising an aqueous dispersion of a pigment.

SOLUTION: This recording fluid comprises an aqueous liquid containing a pigment, colloidal silica and a chelating agent, these ingredients respectively accounting for 0.1–10wt.%, 0.5–30wt.% and 0.01–5wt.% of the recording fluid usually. Examples of the pigment used include quinacridone, phthalocyanine, azo, isoindolinone, dianthraquinonyl red, threne and perylene pigments, and carbon black. An aqueous resin may be used in this recording fluid.

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CLAIMS

[Claim(s)]

[Claim 1] Record liquid for ink jets which contains a pigment, colloidal silica, and a chelating agent in a water liquid, and is characterized by the bird clapper.

[Claim 2] Record liquid for ink jets according to claim 1 which contains a pigment 0.1 to 10% of the weight among record liquid.

[Claim 3] Record liquid for ink jets according to claim 1 or 2 which contains 0.5 – 30 % of the weight, and a chelating agent for colloidal silica 0.01 to 5% of the weight among record liquid.

[Claim 4] Record liquid for ink jets according to claim 1 it is [liquid] one sort as which a pigment is chosen from the group which it becomes from a Quinacridone system, a phthalocyanine system, an azo system, an isoindolinone system, dianthraquinonyl red series, the Indanthrene system, a perylene system, and carbon black.

[Claim 5] furthermore, the claims 1–4 containing an aquosity resin — either — the record liquid for ink jets of a publication

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DETAILED DESCRIPTION

[Detailed Description of the Invention]

[0001]

[The technical field to which invention belongs] this invention relates to the record liquid which is excellent in the water resistance of a printing record object and lightfastness to the so-called regular papers, such as acid paper currently used in general office etc., and alkaline paper, about the record liquid for ink jets, and can perform record of high saturation.

[0002]

[Description of the Prior Art] Conventionally, as record liquid for ink jets, what dissolved water soluble dyes, such as acid dye, direct dye, and basic dye, in a glycol system solvent and water is used well (for example, Provisional Publication No. 53-61412, Provisional Publication No. 54-89811, Provisional Publication No. 55-65269). As a water soluble dye, in order to acquire the stability of record liquid, generally the soluble high thing to water is used. Therefore, the ink-jet record object had the problem of producing bleeding of the color of a record portion easily, when water resistance was bad and generally spilt water.

[0003] In order to improve such a waterproof defect, the structure of a color is changed or to prepare strong basic record liquid is tried (Provisional Publication No. 56-57862). Moreover, aiming at waterproof improvement, using the reaction of the recording paper and record liquid well is also performed (Provisional Publication No. 50-49004, Provisional Publication No. 57-36692, Provisional Publication No. 59-20696, Provisional Publication No. 59-146889). When these methods lack in versatility in that restrictions of the recording paper are received although the effect remarkable about the specific recording paper is obtained and it uses except the specific recording paper, with the record liquid which uses a water soluble dye, sufficient water resistance of a record object is not obtained in many cases.

[0004] Moreover, although there are what distributed or dissolved the oil color in the high boiler, and a thing which dissolved the oil color in the volatile solvent as waterproof good record liquid, there are an odor of a solvent and a problem of discharge of a solvent and it is not desirable on environment. Moreover, there is a problem for which a solvent recovery etc. is needed depending on the installation of the case where a lot of record is performed, or equipment. Then, in order to receive the water resistance of a record object, and lightfastness, development of the record liquid which distributed the pigment to the drainage system medium is performed. In the record liquid for ink jets, the path of a nozzle is becoming thin and the particle diameter of a coloring agent is also turning minutely in connection with this as a printer is expected high resolution. However, it is although the lightfastness more than about the same transparency as a color, clear nature, and a color will become possible in the image formation to transparent base materials, such as an over head projector, if it is made detailed. In the record paper, when pigment-content powder type record liquid (moisture powder ink-jet record liquid) is used to a color dyeing recording paper fiber and a clear picture being acquired, the problem which a pigment falls and cannot conceal the color of the recording paper of a ground and by which clear nature is spoiled between fiber has occurred. This problem is becoming remarkable with detailedization of a pigment. A bad influence is caused to remarkable increase of viscosity, and regurgitation stability although an improvement is carried out to improvement in clear-izing by

the increase in the aquosity resin which is a binder.

[0005]

[Problem(s) to be Solved by the Invention] this invention solves the trouble of the above-mentioned conventional method, and relates to the record liquid for moisture powder ink jets with which transparent and clear quality of image is obtained.

[0006]

[Means for Solving the Problem] this invention relates to the record liquid for ink jets which contains a pigment, colloidal silica, and a chelating agent in a water liquid, and is characterized by the bird clapper. Furthermore, this invention relates to the above-mentioned record liquid for ink jets which contains a pigment 0.1 to 10% of the weight among record liquid. Furthermore, this invention relates colloidal silica to the above-mentioned record liquid for ink jets included 0.01 to 5% of the weight among record liquid in 0.5 – 30 % of the weight, and a chelating agent. Furthermore, this invention relates to the above-mentioned record liquid for ink jets which is chosen from the group which a pigment becomes from a Quinacridone system, a phthalocyanine system, an azo system, an isoindolinone system, dianthraquinonyl red series, the Indanthrene system, a perylene system, and carbon black and whose number is one. Furthermore, this invention relates to the above-mentioned record liquid for ink jets which contains an aquosity resin further.

[0007]

[Embodiments of the Invention] Especially if the pigment used for this invention does not dissolve in water, it is not limited. As an insoluble color, that which insolubilized a disperse dye, the water-insoluble nature color of the oil color or direct dye, acid dye, basic dye, etc. by lake-ization is also used. However, the color which dissolves in the synthetic resin to be used completely is excepted from this invention.

[0008] As an example of a pigment, toluidine red, toluidine MARUN, a Hansa Yellow, Insoluble azo pigment, such as benzidine yellow and pyrazolone red, a Lithol Red, Soluble azo pigment, such as helio bordeaux, a pigment scarlet, and Permanent-Red 2B, Phthalocyanine systems, such as a copper phthalocyanine blue and a Phthalocyanine Green, Quinacridone systems, such as Quinacridone red and a Quinacridone Magenta, There are isoindolinone systems, such as perylene systems, such as perylene red and perylene Scarlett, isoindolinone yellow, and an isoindolinone orange, dianthraquinonyl red series, the Indanthrene system guided from a vat dye, carbon black, etc.

[0009] The following pigment can be illustrated as such a pigment. A Color Index (C. I.) number shows. C. I. pigment yellow 12, 13, 14, 17, 20, 24, 74, 83, and 86 93, 109, 110, 117, 125, 137, 138, 147, 148, 153, 154, 166, 161, the C.I. pigment oranges 13, 16, 36, 43, and 51, 55, 59, 61, the C.I. pigment red 9, 48, 49, 52, 53, 57, 97, 122, 123, 149, 168, 177, 180, 192, 215, 216, 217, 220, 223, 224, 226, 227, 228, 238, and 240, C. I. pigment violet 19, 23, 29, 30, 37, 40, and 50, the C.I. pigment blues 15, 15:1, 15:4, 15:6, 22, 60, and 64, the C.I. pigment greens 7 and 36, C.I. pigment Brown 23, 25, and 26, C. I. pigment black 6 and 7 grades can be illustrated.

[0010] As the diameter of a particulate material and the diameter of a primary particle of a desirable processing pigment, 1.0 micrometers or less of mean particle diameters are 0.2 micrometers or less still more preferably in the measurement or the electron microscope by laser dispersion. When the diameter of a particulate material and primary particle of a pigment are 0.2 micrometers or more, you may carry out salt milling processing (processing which makes mineral salt a grinding assistant and makes the diameter of a primary particle of a pigment detailed mechanically, using a little solvent as a binder) for a pigment. Moreover, you may use the surface treatment pigment to which the functional group was made to add on the surface of a pigment. Filtration operation of an in [that it is such a particle size / manufacture of record liquid] is easy, and sedimentation's by the passage of time of record liquid decreases.

[0011] The anion of a silanol group and a hydroxyl group exists on the surface of a particle, and the colloidal silica in this invention makes the ultrafine particle of a silicic acid anhydride the colloidal solution, and contains and stabilizes soda as a sodium oxide, and the silica with the size (particle size of 1–100nm) of colloid is distributing it stably in a water liquid. The colloidal silica used in this invention uses the thing of the particle diameter of the size of the range of 5–

100nm. It is 5–60nm to which it is desirable, a particle diameter is fine, and particle size distribution were equal. Moreover, two or more kinds from which a particle diameter differs may be used together, pH regulator can be adjusted suitably if needed, and a basic component or an acidic component can also be added. Specifically, snow tex (tradename by the Nissan chemistry company), RUDOKKUSU (Du Pont tradename), KATAROIDO (tradename by the catalyst chemistry company), an ardealite (tradename by the Asahi electrification company), etc. are mentioned.

[0012] The weight ratio of colloidal silica is 1 – 15 % of the weight more preferably 0.5 to 30% of the weight in composition of record liquid. If there are few these amounts, the good ink of clear nature will not be obtained, and on the other hand, if this amount is large, ink with good preservation stability and **** stability will not be obtained.

[0013] The chelating agent in this invention reacts with a metal ion, and builds a chelate ring. Ring formation happens by generation of covalent bond, coordinate bond, or its both sides. Since the effect of a chelating agent is in the supplement of a metal ion, it supplements with the metal ion of the front face of the colloidal silica contained in record liquid. For this reason, colloidal silica will be in a metastable state, it is condensed on space, and builds a film, its depression of coloring matter decreases, and its clear nature improves. However, if a chelating agent is not included, the clear nature at the time of considering as the record liquid of the alkali field which does not make a head corrode will fall.

[0014] As a ligand which had two acidic groups as an example of a chelating agent, there are a malonic acid, oxalic acid, a phthalic acid, a glycolic acid, and a salicylic acid, for example. As a ligand with one acidic group and one un-acid coordination machine, there are an eight quinolinol, an acetylacetone, a truffe RUORO acetone, a dimethylglyoxime, a dithizone, and a salichlaldehyde. There are an ethylenediamine, 2, and 2'-bipyridine, 1, and a 10-phenanthroline as a ligand with two un-acid coordination machines. Ethylenediaminetetraacetic acid, a propylenediamine tetraacetic acid, a butylene diamine tetraacetic acid, pentylene diamine tetraacetic acids and these sodium salt, or an ammonium salt is mentioned as an amino polycarboxylic acid. In composition of the record liquid for ink jets, the weight ratio of a chelating agent is 0.01 – 5 % of the weight, and is 0.1 – 1 % of the weight preferably. If there are few amounts of a chelating agent than a minimum, the good ink of clear nature will not be obtained, and if [than an upper limit] more, ink with good preservation stability and regurgitation stability will not be obtained.

[0015] It is more desirable to use the surfactant of anionic or Nonion nature, in order to have the performance which was especially excellent in transparency, clear nature, and distribution stability, although a surfactant may be used as a dispersant in order to distribute a pigment stably, without precipitating in the record liquid for ink jets. Colloidal silica is for condensing when the dispersant for the front face of colloidal silica being charged in minus as a reason using anionic or Nonion nature as a surfactant, and stabilizing a pigment is cation nature. As an anionic surfactant, a fatty-acid salt, an alkyl-sulfuric-acid ester salt, Alkylaryl sulfonates, alkylnaphthalenesulfonate, A dialkyl sulfonate, a dialkyl sulfo succinic-acid salt, alkyl diaryl ether disulfon acid chloride, Alkyl phosphate, a polyoxyethylene-alkyl-ether sulfate, a polyoxyethylene-alkyl-aryl-ether sulfate, A naphthalene sulfonic-acid formalin condensate, polyoxyethylene alkyl phosphate, glycerol borate fatty acid ester, polyoxyethylene glycerol fatty acid ester, etc. can be illustrated.

[0016] As a Nonion nature surfactant, polyoxyethylene alkyl ether, polyoxyethylene alkyl aryl ether, a polyoxyethylene oxypropylene block copolymer, a sorbitan fatty acid ester, a polyoxy EKIREN sorbitan fatty acid ester, polyoxyethylene sorbitol fatty acid ester, a glycerine fatty acid ester, polyoxyethylene fatty acid ester, polyoxyethylene alkylamine, a fluorine system, a silicon system, etc. can be illustrated.

[0017] Although are not limited to especially the amount of the surfactant used, and 0.1 – 15% of the weight of the range is generally desirable to the total weight of ink, it is 0.1 – 10 % of the weight still more preferably.

[0018] It is desirable especially desirable especially for making a printing recording characteristic good the distributed stability of record liquid and that pH of a water liquid is 7–10, and the record

liquid for ink jets of this invention is the weak alkalinity of 7-9. When pH is six or less, the stability of record liquid produces condensation or gelling in the passage of time preferably, and the stability of colloidal silica is bad at ten or more. An amine, mineral salt, ammonia, a phosphoric acid, an acetic acid, etc. can be used for adjustment of pH.

[0019] In the record liquid for ink jets of this invention, the water solvent used besides water prevents dryness in the nozzle portion of record liquid, and individual-ization of record liquid, and prevents injection of stable record liquid, and dryness by the passage of time of a nozzle. As such a water solvent, ethylene glycol, a diethylene glycol, A propylene glycol, a triethylene glycol, a triethylene glycol, A polyethylene glycol, a glycerol, a tetraethylene glycol, A dipropylene glycol, ketone alcohol, the diethylene-glycol monobutyl ether, Ethylene-glycol-monobutyl-ether ethylene glycol monoethyl ether, 1, 2-hexandiol, a N-methyl-2-pyrrolidone, a substitution pyrrolidone, 2 and 4, 6-hexane triol, tetrafurfuryl alcohol, 4-methoxy pentane non, etc. can be illustrated.

[0020] In the record liquid for ink jets of this invention, in order to fix a pigment good, you may use an aquosity resin. As for this aquosity resin, the resin or the water-dispersion resin of dissolved water in fuel is used. As such an aquosity resin, there are a dissolved-water-in-fuel resin or water-dispersion resins, such as acrylic, styrene - acrylic, a polyester system, a polyamide system, and a polyurethane system. Making 0.5 - 10 % of the weight contain in the record liquid for ink jets uses these resins one to 5% of the weight desirable still more preferably. If fewer than 0.5 % of the weight, a coloring agent cannot fully be established. Moreover, when it increases more than 10 % of the weight, the regurgitation stability of record liquid may be reduced. In addition, although there is an inclination which makes viscosity of record liquid high when the resin of dissolved water in fuel is used as an aquosity resin, by the water-dispersion resin, viscosity can stop low and can improve the water resistance of a record object. If needed, neutralizers, such as ammonia, an amine, and inorganic alkali, can be adjusted suitably, and these resins can add them.

[0021] Moreover, since an apparent drying property is early carried out in the purpose which speeds up the dryness to the paper of record liquid, a penetrating agent can be added. Glycol ethers, such as a methanol, ethanol, isopropyl alcohol, and the diethylene-glycol monobutyl ether, alkylene glycol, an alkylene diol, the polyethylene-glycol monochrome lauryl ether, a sodium lauryl sulfate, sodium dodecylbenzenesulfonate, a sodium oleate, sodium dioctyl sulfosuccinate, etc. can be used as such a penetrating agent. When these have effect sufficient by the amount of 5 or less % of the weight used of record liquid and there are than this, they become less desirable [start a blot of printing and a paper omission (print through) and].

[more]

[0022] An antifungal agent can also be added in order to prevent generating of mold in the record liquid of this invention. As for a concrete target, the amine salt of a sodium dehydroacetate, a sodium benzoate, sodium pyridine thione-1-oxide, zinc pyridine thione-1-oxide, 1, 2-bends iso thiazoline-3-ON, and 1-bends iso thiazoline-3-ON etc. is used. these — 0.05— of record liquid — it can use about 1.0% of the weight Moreover, since generating of the bubble at the time of circulation of record liquid or movement, and manufacture of record liquid is prevented, a defoaming agent can also be added.

[0023] It is possible to use it as a color, as long as there is no problem in water resistance and lightfastness for the purpose of adjustment of the hue of a pigment, grant of concentration, etc. since stability of distribution of a pigment may be worsened by use of a color — a pigment — it is necessary to stop to 25 or less % of the weight of use preferably 40 or less % of the weight As a color, that which insolubilized a disperse dye, the water-insoluble nature color of the oil color, direct dye, acid dye, basic dye, etc. by lake-ization, a reactive dye, a metallized dye, etc. are used. These colors have the desirable refining color from which mineral salt was removed.

[0024] As an example of a color C. The I. direct blacks 17, 19, 32, 51, 71, 108, 146, 154, and 166, the C.I. ADDO blacks 2, 7, 24, 26, 31, 52, 63, 112, and 118, the C.I. basic black 2, C.I. direct blues 6, 22, 25, and 71, 90, 106, the C.I. acid blues 9, 22, 40, 59, 93, 102, 104, 113, 117, 120, 167, 229, and 234, the C.I. basic blues 1, 3, 5, 7, 9, 24, 25, 26, 28, and 29, the C.I. direct red 1, 4, and 17, 28, 83, the C.I. acid reds 1, 6, 32, 37, 51, 52, 80, 85, 87, 92, 94, 115, 180, 256, and 315, the 317.C.I.

basic red 1, 2, 9, 12, 13, 14, and 37, the C.I. direct yellow 12, 24, and 26, 98, the C.I. acid yellow 11, 17, 23, 25, 29, 42, 61, and 71, the C.I. basic yellow 11, the 28.C.I. direct oranges 34, 39, 44, 46, and 60, the C.I. direct violet 47 and 48, C. I. direct Brown 109, the C.I. direct green 59, the C.I. acid oranges 7 and 19, the C.I. acid violet 49, the C.I. basic violet 7 and 14, and 27 grades can be illustrated. A urea, a dimethyl urea, etc. can also be added as other additives.

[0025] The dispersing element of the pigment detailed—ization—processed about manufacture of record liquid, for example, a dispersant, [whether it distributes in a sand mill, a homogenizer, a ball mill, a paint shaker, an ultrasonic disperser, etc. by mixing water or an aquosity resin, an aquosity solvent, etc., and] Or after kneading beforehand well in 2 roll mills, it distributes further in the above—mentioned sand mill etc., dilution, colloidal silica, a chelating agent, and other additives are mixed with water suitably, and record liquid is manufactured. A high—speed disperser besides stirring by the agitator using the usual feather, an emulsifier, etc. can perform mixed stirring.

[0026] Record liquid is enough filtered with the filter of 3 micrometers or less of apertures before dilution or to the back. It is desirable to filter with a filter 0.45 micrometers or less still more preferably with a filter 1.0 micrometers or less preferably. In advance of filtration of a filter, by centrifugal separation, the thing of a big particle size can also be removed, blinding in filtration with a filter is lessened by this, and the duration of service of a filter becomes long.

[0027] Although record liquid is based also on the method of a recording device, it is desirable to adjust as a liquid of viscosity 0.8 – 15 mPa·s (25 degrees C). 25 – 60 dyn/cm of surface tension is desirable, and the weak alkalinity of pH of 7–9 is desirable.

[0028] Though it is aquosity, since the record liquid manufactured by this invention has remarkably good water resistance, it is suitably used as record liquid for ink jets, and it can also be used for the field of record objects, such as preparation in office, a sign, marking of the corrugated paper used for an out board use since lightfastness is further superior to the color type, numbering, and a bar code.

[0029]

[Example] Hereafter, this invention is explained based on an example. That it is with the example central part and % shows weight section and weight %, respectively.

Example 1 [the creation method of a salt milling processing pigment]

Crude copper phthalocyanine (Toyo Ink make) 250 section sodium chloride 2500 section diethylene glycol The 200 sections were taught to the 1 gallon kneader of stainless steel, and were kneaded for 3 hours. Next, this mixture was supplied to 2.5l. warm water, and it heated and stirred at about 80 degrees C. After stirring for about 1 hour and considering as the shape of a slurry, filtration and rinsing were repeated 5 times and the watercolor—pigment dispersing element (salt milling processing article) of 50% of solid contents was obtained except for the sodium chloride and the solvent. The following raw material was paid to the sand mill, it distributed for 3 hours, and the concentration record liquid for ink jets was created.

[0030]

[The creation method of dark color record liquid]

Indigo pigment (50% of salt milling processing article solid contents) . Section [30.0 /]

Dimethylamino Ethanol The 0.1 sections Dispersant (emulgen 420 by Kao Corp.) The 8.0 sections Purified water The 55.7 sections Glycerol The 6.0 sections Sodium OMAJIN (aurin company make) It mixed with the following after 0.2 partial powder. It filtration and filtered with the 0.45—micrometer membrane filter continuously by the 1—micrometer membrane filter after mixture, and record liquid was manufactured.

[0031]

[The creation method of record liquid]

The above—mentioned concentration record liquid . Section [16.7 /] Colloidal Silica (Snow Tex 30 by Nissan Chemistry Company) The 16.7 sections Dimethylamino ethanol The 0.1 sections Dispersant (emulgen 420 by Kao Corp.) The 1.2 sections Acrylic resin (Japanese polymer company make W-215) The 3.0 sections Sodium OMAJIN (aurin company make) The 0.2 sections Ethylenediaminetetraacetic acid sodium salt The 0.1 sections Glycerol The 10.0 sections Purified water The 52.0 sections. [0032] The filterability when obtaining record liquid was evaluated,

viscosity and the mean particle diameter were evaluated as follows about the obtained record liquid, and the clear nature of the injection property when using this record liquid further, a printing state, and a record object, transparency, the water resistance of a record object, etc. were evaluated. A result is shown in Table 1.

The amount currently filtered within [filterable] fixed time with the membrane filter (1 micrometer and 0.45 micrometers) with a diameter of 90mm.

It measured at 25 degrees C using [viscosity] Brookfield viscometer.

It measured with the particle-size-distribution plan ("SALD-1100" by Shimadzu Corp.) of a [mean-particle-diameter] laser diffraction method.

[Printing state] record liquid was put into the cartridge of Epson HG5130, and it recorded on the regular paper (Xerox Corp. make K). Visual evaluation of the printing state of a record object was carried out.

The injection state of the nozzle under [injection property] continuation printing was evaluated with the printing object.

Good: Continuation printing has been correctly carried out in a predetermined position.

Defect: In spite of having carried out continuation printing, a deficit is not produced on the way or it is not printed in a predetermined position.

When it recorded on a [waterproof] regular paper (Xerox Corp. make K), water was hung down to the recording surface or viewing estimated the existence of bleeding of the ink when being under water for 1 minute, and the flow broth of ink after drying what was printed.

Viewing estimated the transparency when carrying out drawdown of the ink to the OHP sheet for [transparency] ink jets, and making it it in a 1.5-mil applicator.

Drawdown was carried out to the [clear nature] regular paper (Xerox Corp. make K), and both C values of Munsell of viewing and a color machine machine (Nippon Denshoku Industries make sigma 80) compared clear nature.

Drawdown was carried out to the [light-fast] regular paper (Xerox Corp. make K), the accelerated test was performed in the fadeometer (ultraviolet-rays long life fadeometer by the SUGA testing-machine company), and both delta E value of viewing and a color machine machine (Nippon Denshoku Industries make sigma 80) compared the grade of the tenebrescence of a color.

[0033] Instead of the crude copper phthalocyanine of example 2 example 1, the Hansa-Yellow system yellow pigment (HOSUTA palm yellow H-3 by Hoechst A.G. G) 260 section was used, and the aquosity dispersing element (salt milling processing article) was obtained like the example 1. Then, the concentration record liquid for ink jets was created like the example 1. The record liquid for ink jets excellent in transparency and clear nature was obtained. An evaluation result is shown in Table 1.

[0034] Instead of the crude copper phthalocyanine of example 3 example 1, the Quinacridone system red-pigments (HOSUTA palm pink E by Hoechst A.G.) 250 section was used, and the aquosity dispersing element (salt milling processing article) was obtained like the example 1. Then, the concentration record liquid for ink jets was created like the example 1. The record liquid for ink jets excellent in transparency and clear nature was obtained. An evaluation result is shown in Table 1.

[0035] Instead of the crude copper phthalocyanine of example 4 example 1, the iso indoline system yellow pigment (PARIO toll yellow D-1819 by BASF A.G.) was used. The concentration record liquid for ink jets was created by the same method as an example 1, with the SORUTOMI link of an example 1 not performed. The record liquid for ink jets excellent in transparency and clear nature was obtained. An evaluation result is shown in Table 1.

[0036] Instead of the crude copper phthalocyanine of example 5 example 1, diamino JIAN truck nil system red pigments (chlromophtal red made from Ciba-Geigy A-2B) were used. The salt milling of an example 1 created the concentration record liquid for ink jets by the same method as an example 1, not carrying out. The record liquid for ink jets excellent in transparency and clear nature was obtained. An evaluation result is shown in Table 1.

[0037] Instead of the crude copper phthalocyanine of example 6 example 1, the indanthrene system blue-pigment (Toyo Ink RIONO gene blue 6505) 250 section was used, and the aquosity

dispersing element (salt milling processing article) was obtained like the example 1. Then, the concentration record liquid for ink jets was created like the example 1. The record liquid for ink jets excellent in transparency and clear nature was obtained. An evaluation result is shown in Table 1.

[0038] Without carrying out the same salt milling down stream processing as an example 1 using example 7 carbon black, the following raw material was paid to the sand mill, it distributed for 3 hours, and the concentration record liquid for ink jets was created.

Carbon black (Mitsubishi Chemical # 2600) The 15.0 sections Dimethylamino ethanol The 0.1 sections Dispersant (emulgen 420 by Kao Corp.) The 8.0 sections Purified water The 70.7 sections Glycerol The 6.0 sections Make it be the same as that of an example 1 after that [sodium OMAJIN (product made from aurin) 0.2 section]. The record liquid for ink jets was created. The record liquid for ink jets excellent in transparency and clear nature was obtained. An evaluation result is shown in Table 1.

[0039] What compensated with the insufficiency by the purified water except for colloidal silica and the chelating agent at the time of manufacture of record liquid about the pigment used in the one to example of comparison 7 examples 1-7 was used as the concentration record liquid for ink jets of the examples 1-7 of comparison, respectively. Evaluation is performed like an example and a result is shown in Table 2.

the examples 8-11 of comparison — the result which performed evaluation like the example about a certain color type of record liquid (YMCBK) for ink jets is shown in Table 3

[0040] In addition, when the natural dissolution of the record liquid obtained in the examples 1-7 and the record liquid obtained in the examples 1-7 of comparison is carried out after one-week preservation and it is saved by the 60-degree C thermostat at -40 degrees C in January, it holds at -40 degrees C for 3 hours, and applies for 3 hours, and it is to 60 degrees C. When the temperature up was carried out, and it maintenance and cooled to -40 degrees C over 3 hours for 3 hours continuously at 60 degrees C and this was repeated for three days, in the case of which, there is also no generating of settling, early viscosity was maintained, and the injection property was also stable.

[0041]

[Table 1]

	実施例 1	実施例 2	実施例 3	実施例 4	実施例 5	実施例 6	実施例 7
透過性 1μ $0, 45 \mu$	○ ○						
粘度 cps	3. 0	3. 5	3. 2	2. 8	2. 5	3. 0	2. 5
平均粒径 μm	100	120	110	125	90	100	90
印字状態	◎	◎	◎	◎	◎	◎	◎
噴射特性	○	○	○	○	○	○	○
耐水性	○	○	○	○	○	○	○
透明性	○	○	○	○	○	○	○
鮮明性	○	○	○	○	○	○	○
耐光性	○	○	○	○	○	○	○

[0042]
[Table 2]

	比較例 1	比較例 2	比較例 3	比較例 4	比較例 5	比較例 6	比較例 7
滲透性 1μ 0.45μ	○ ○						
粘度 cps	3.0	3.5	3.2	2.8	2.5	3.0	2.5
平均粒径 μm	100	120	110	125	90	100	90
印字狀態	○	○	○	○	○	○	○
噴射特性	○	○	○	○	○	○	○
耐水性	○	○	○	○	○	○	○
透明性	○	○	○	○	○	○	○
鮮明性	×	×	×	×	×	×	×
耐光性	○	○	○	○	○	○	○

[0043]
[Table 3]

	比較例 8 Y	比較例 9 M	比較例11 C	比較例12 BK
濾過性 1μ	○	○	○	○
0.45μ	○	○	○	○
粘度 c p s	3.1	3.2	3.1	3.0
平均粒径 μm	—	—	—	—
印字狀態	◎	◎	◎	◎
噴射特性	○	○	○	○
耐水性	×	×	×	×
透明性	○	○	○	○
鮮明性	○	○	○	○
耐光性	×	×	×	×

[Effect of the Invention] The record liquid for ink jets which can enable high-definition printing which a printing record object has sufficient water resistance and sufficient lightfastness, and is satisfactory also to long-term preservation, and is further satisfactory also to blinding was able to be offered without having a bad influence on the picture grace of a record object, when this invention performs printing record to the so-called regular papers, such as acid paper currently used in general office etc., and alkaline paper.

[Translation done.]

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TECHNICAL FIELD

[The technical field to which invention belongs] this invention relates to the record liquid which is excellent in the water resistance of a printing record object and lightfastness to the so-called regular papers, such as acid paper currently used in general office etc., and alkaline paper, about the record liquid for ink jets, and can perform record of high saturation.

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PRIOR ART

[Description of the Prior Art] Conventionally, as record liquid for ink jets, what dissolved water soluble dyes, such as acid dye, direct dye, and basic dye, in a glycol system solvent and water is used well (for example, Provisional Publication No. 53-61412, Provisional Publication No. 54-89811, Provisional Publication No. 55-65269). As a water soluble dye, in order to acquire the stability of record liquid, generally the soluble high thing to water is used. Therefore, the ink-jet record object had the problem of producing bleeding of the color of a record portion easily, when water resistance was bad and generally spilt water.

[0003] In order to improve such a waterproof defect, the structure of a color is changed or to prepare strong basic record liquid is tried (Provisional Publication No. 56-57862). Moreover, aiming at waterproof improvement, using the reaction of the recording paper and record liquid well is also performed (Provisional Publication No. 50-49004, Provisional Publication No. 57-36692, Provisional Publication No. 59-20696, Provisional Publication No. 59-146889). When these methods lack in versatility in that restrictions of the recording paper are received although the effect remarkable about the specific recording paper is obtained and it uses except the specific recording paper, with the record liquid which uses a water soluble dye, sufficient water resistance of a record object is not obtained in many cases.

[0004] Moreover, although there are what distributed or dissolved the oil color in the high boiler, and a thing which dissolved the oil color in the volatile solvent as waterproof good record liquid, there are an odor of a solvent and a problem of discharge of a solvent and it is not desirable on environment. Moreover, there is a problem for which a solvent recovery etc. is needed depending on the installation of the case where a lot of record is performed, or equipment. Then, in order to receive the water resistance of a record object, and lightfastness, development of the record liquid which distributed the pigment to the drainage system medium is performed. In the record liquid for ink jets, the path of a nozzle is becoming thin and the particle diameter of a coloring agent is also turning minutely in connection with this as a printer is expected high resolution. However, it is although the lightfastness more than about the same transparency as a color, clear nature, and a color will become possible in the image formation to transparent base materials, such as an over head projector, if it is made detailed. In the record paper, when pigment-content powder type record liquid (moisture powder ink-jet record liquid) is used to a color dyeing recording paper fiber and a clear picture being acquired, the problem which a pigment falls and cannot conceal the color of the recording paper of a ground and by which clear nature is spoiled between fiber has occurred. This problem is becoming remarkable with detailedization of a pigment. A bad influence is caused to remarkable increase of viscosity, and **** stability although an improvement is carried out to improvement in clear-izing by the increase in the water resin which is a binder.

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EFFECT OF THE INVENTION

[Effect of the Invention] The record liquid for ink jets which can enable high-definition printing which a printing record object has sufficient water resistance and sufficient lightfastness, and is satisfactory also to long-term preservation, and is further satisfactory also to blinding was able to be offered without having a bad influence on the picture grace of a record object, when this invention performs printing record to the so-called regular papers, such as acid paper currently used in general office etc., and alkaline paper.

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TECHNICAL PROBLEM

[Problem(s) to be Solved by the Invention] this invention solves the trouble of the above-mentioned conventional method, and relates to the record liquid for moisture powder ink jets with which transparent and clear quality of image is obtained.

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MEANS

[Means for Solving the Problem] this invention relates to the record liquid for ink jets which contains a pigment, colloidal silica, and a chelating agent in a water liquid, and is characterized by the bird clapper. Furthermore, this invention relates to the above-mentioned record liquid for ink jets which contains a pigment 0.1 to 10% of the weight among record liquid. Furthermore, this invention relates colloidal silica to the above-mentioned record liquid for ink jets included 0.01 to 5% of the weight among record liquid in 0.5 – 30 % of the weight, and a chelating agent. Furthermore, this invention relates to the above-mentioned record liquid for ink jets which is chosen from the group which a pigment becomes from a Quinacridone system, a phthalocyanine system, an azo system, an isoindolinone system, dianthraquinonyl red series, the Indanthrene system, a perylene system, and carbon black and whose number is one. Furthermore, this invention relates to the above-mentioned record liquid for ink jets which contains a water resin further.

[0007]

[Embodiments of the Invention] Especially if the pigment used for this invention does not dissolve in water, it is not limited. As an insoluble color, that which insolubilized a disperse dye, the water-insoluble nature color of the oil color or direct dye, acid dye, basic dye, etc. by lake-ization is also used. However, the color which dissolves in the synthetic resin to be used completely is excepted from this invention.

[0008] As an example of a pigment, toluidine red, toluidine MARUN, a Hansa Yellow, Insoluble azo pigment, such as benzidine yellow and pyrazolone red, a Lithol Red, Soluble azo pigment, such as helio bordeaux, a pigment scarlet, and Permanent-Red 2B, Phthalocyanine systems, such as a copper phthalocyanine blue and a Phthalocyanine Green, Quinacridone systems, such as Quinacridone red and a Quinacridone Magenta, There are isoindolinone systems, such as perylene systems, such as perylene red and perylene Scarlett, isoindolinone yellow, and an isoindolinone orange, dianthraquinonyl red series, the Indanthrene system guided from a vat dye, carbon black, etc.

[0009] The following pigment can be illustrated as such a pigment. A Color Index (C. I.) number shows. C. I. pigment yellow 12, 13, 14, 17, 20, 24, 74, 83, and 86 93, 109, 110, 117, 125, 137, 138, 147, 148, 153, 154, 166, 161, the C.I. pigment oranges 13, 16, 36, 43, and 51, 55, 59, 61, the C.I. pigment red 9, 48, 49, 52, 53, 57, 97, 122, 123, 149, 168, 177, 180, 192, 215, 216, 217, 220, 223, 224, 226, 227, 228, 238, and 240, C. I. pigment violet 19, 23, 29, 30, 37, 40, and 50, the C.I. pigment blues 15, 15:1, 15:4, 15:6, 22, 60, and 64, the C.I. pigment greens 7 and 36, C.I. pigment Brown 23, 25, and 26, C. I. pigment black 6 and 7 grades can be illustrated.

[0010] As the diameter of a particulate material and the diameter of a primary particle of a desirable processing pigment, 1.0 micrometers or less of mean particle diameters are 0.2 micrometers or less still more preferably in the measurement or the electron microscope by laser dispersion. When the diameter of a particulate material and primary particle of a pigment are 0.2 micrometers or more, you may carry out salt milling processing (processing which makes mineral salt a grinding assistant and makes the diameter of a primary particle of a pigment detailed mechanically, using a little solvent as a binder) for a pigment. Moreover, you may use the surface treatment pigment to which the functional group was made to add on the surface of a

pigment. Filtration operation of an in [that it is such a particle size / manufacture of record liquid] is easy, and sedimentation's by the passage of time of record liquid decreases.

[0011] The anion of a silanol group and a hydroxyl group exists on the surface of a particle, and the colloidal silica in this invention makes the ultrafine particle of a silicic acid anhydride the colloidal solution, and contains and stabilizes soda as a sodium oxide, and the silica with the size (particle size of 1–100nm) of colloid is distributing it stably in a water liquid. The colloidal silica used in this invention uses the thing of the particle diameter of the size of the range of 5–100nm. It is 5–60nm to which it is desirable, a particle diameter is fine, and particle size distribution were equal. Moreover, two or more kinds from which a particle diameter differs may be used together, pH regulator can be adjusted suitably if needed, and a basic component or an acidic component can also be added. Specifically, snow tex (tradename by the Nissan chemistry company), RUDOKKUSU (Du Pont tradename), KATAROIDO (tradename by the catalyst chemistry company), an ardealite (tradename by the Asahi electrification company), etc. are mentioned.

[0012] The weight ratio of colloidal silica is 1 – 15 % of the weight more preferably 0.5 to 30% of the weight in composition of record liquid. If there are few these amounts, the good ink of clear nature will not be obtained, and on the other hand, if this amount is large, ink with good preservation stability and regurgitation stability will not be obtained.

[0013] The chelating agent in this invention reacts with a metal ion, and builds a chelate ring. Ring formation happens by generation of covalent bond, coordinate bond, or its both sides. Since the effect of a chelating agent is in the supplement of a metal ion, it supplements with the metal ion of the front face of the colloidal silica contained in record liquid. For this reason, colloidal silica will be in a metastable state, it is condensed on space, and builds a film, its depression of coloring matter decreases, and its clear nature improves. However, if a chelating agent is not included, the clear nature at the time of considering as the record liquid of the alkali field which does not make a head corrode will fall.

[0014] As a ligand which had two acidic groups as an example of a chelating agent, there are a malonic acid, oxalic acid, a phthalic acid, a glycolic acid, and a salicylic acid, for example. As a ligand with one acidic group and one un-acid coordination machine, there are an eight quinolinol, an acetylacetone, a truffe RUORO acetone, a dimethylglyoxime, a dithizone, and a salichlaldehyde. There are an ethylenediamine, 2, and 2'-bipyridine, 1, and a 10-phenanthroline as a ligand with two un-acid coordination machines. Ethylenediaminetetraacetic acid, a propylenediamine tetraacetic acid, a butylene diamine tetraacetic acid, pentylene diamine tetraacetic acids and these sodium salt, or an ammonium salt is mentioned as an amino polycarboxylic acid. In composition of the record liquid for ink jets, the weight ratio of a chelating agent is 0.01 – 5 % of the weight, and is 0.1 – 1 % of the weight preferably. If there are few amounts of a chelating agent than a minimum, the good ink of clear nature will not be obtained, and if [than an upper limit] more, ink with good preservation stability and regurgitation stability will not be obtained.

[0015] It is more desirable to use the surfactant of anionic or Nonion nature, in order to have the performance which was especially excellent in transparency, clear nature, and distribution stability, although a surfactant may be used as a dispersant in order to distribute a pigment stably, without precipitating in the record liquid for ink jets. Colloidal silica is for condensing when the dispersant for the front face of colloidal silica being charged in minus as a reason using anionic or Nonion nature as a surfactant, and stabilizing a pigment is cation nature. As an anionic surfactant, a fatty-acid salt, an alkyl-sulfuric-acid ester salt, Alkylaryl sulfonates, alkylnaphthalenesulfonate, A dialkyl sulfonate, the dialkyl sulfo succinate, alkyl diaryl ether disulfon acid chloride, Alkyl phosphate, a polyoxyethylene-alkyl-ether sulfate, a polyoxyethylene-alkyl-aryl-ether sulfate, A naphthalene sulfonic-acid formalin condensate, a polyoxyethylene alkyl phosphate, glycerol borate fatty acid ester, polyoxyethylene glycerol fatty acid ester, etc. can be illustrated.

[0016] As a Nonion nature surfactant, polyoxyethylene alkyl ether, polyoxyethylene alkyl aryl ether, a polyoxyethylene oxypropylene block copolymer, a sorbitan fatty acid ester, a polyoxy EKIREN sorbitan fatty acid ester, polyoxyethylene sorbitol fatty acid ester, a glycerine fatty acid

ester, polyoxyethylene fatty acid ester, polyoxyethylene alkylamine, a fluorine system, a silicon system, etc. can be illustrated.

[0017] Although are not limited to especially the amount of the surfactant used, and 0.1 – 15% of the weight of the range is generally desirable to the total weight of ink, it is 0.1 – 10 % of the weight still more preferably.

[0018] It is desirable especially desirable especially for making a printing recording characteristic good the distributed stability of record liquid and that pH of a water liquid is 7–10, and the record liquid for ink jets of this invention is the weak alkalinity of 7–9. When pH is six or less, the stability of record liquid produces condensation or gelling in the passage of time preferably, and the stability of colloidal silica is bad at ten or more. An amine, mineral salt, ammonia, a phosphoric acid, an acetic acid, etc. can be used for adjustment of pH.

[0019] In the record liquid for ink jets of this invention, the aquosity solvent used besides water prevents dryness in the nozzle portion of record liquid, and individualization of record liquid, and prevents injection of stable record liquid, and dryness by the passage of time of a nozzle. As such an aquosity solvent, ethylene glycol, a diethylene glycol, A propylene glycol, a triethylene glycol, a triethylene glycol, A polyethylene glycol, a glycerol, a tetraethylene glycol, A dipropylene glycol, the ketone alcohol, the diethylene-glycol monobutyl ether, Ethylene-glycol-monobutyl-ether ethylene glycol monoethyl ether, 1, 2-hexandiol, a N-methyl-2-pyrrolidone, a substitution pyrrolidone, 2 and 4, 6-hexane triol, tetrafurfuryl alcohol, 4-methoxy pentane non, etc. can be illustrated.

[0020] In the record liquid for ink jets of this invention, in order to fix a pigment good, you may use an aquosity resin. As for this aquosity resin, the resin or the water-dispersion resin of dissolved water in fuel is used. As such an aquosity resin, there are a dissolved-water-in-fuel resin or water-dispersion resins, such as acrylic, styrene – acrylic, a polyester system, a polyamide system, and a polyurethane system. Making 0.5 – 10 % of the weight contain in the record liquid for ink jets uses these resins one to 5% of the weight desirable still more preferably. If fewer than 0.5 % of the weight, a coloring agent cannot fully be established. Moreover, when it increases more than 10 % of the weight, the regurgitation stability of record liquid may be reduced. In addition, although there is an inclination which makes viscosity of record liquid high when the resin of dissolved water in fuel is used as an aquosity resin, by the water-dispersion resin, viscosity can stop low and can improve the water resistance of a record object. If needed, neutralizers, such as ammonia, an amine, and inorganic alkali, can be adjusted suitably, and these resins can add them.

[0021] Moreover, since an apparent drying property is early carried out in the purpose which speeds up the dryness to the paper of record liquid, a penetrating agent can be added. Glycol ethers, such as a methanol, ethanol, isopropyl alcohol, and the diethylene-glycol monobutyl ether, alkylene glycol, an alkylene diol, the polyethylene-glycol monochrome lauryl ether, a sodium lauryl sulfate, sodium dodecylbenzenesulfonate, a sodium oleate, sodium dioctyl sulfosuccinate, etc. can be used as such a penetrating agent. When these have effect sufficient by the amount of 5 or less % of the weight used of record liquid and there are than this, they become less desirable [start a blot of printing and a paper omission (print through) and].

[more]

[0022] An antifungal agent can also be added in order to prevent generating of mold in the record liquid of this invention. As for a concrete target, the amine salt of a sodium dehydroacetate, a sodium benzoate, sodium pyridine thione-1-oxide, zinc pyridine thione-1-oxide, 1, 2-bends iso thiazoline-3-ON, and 1-bends iso thiazoline-3-ON etc. is used. these — 0.05– of record liquid — it can use about 1.0% of the weight Moreover, since generating of the bubble at the time of circulation of record liquid or movement, and manufacture of record liquid is prevented, a defoaming agent can also be added.

[0023] It is possible to use it as a color, as long as there is no problem in water resistance and lightfastness for the purpose of adjustment of the hue of a pigment, grant of concentration, etc. since stability of distribution of a pigment may be worsened by use of a color — a pigment — it is necessary to stop to 25 or less % of the weight of use preferably 40 or less % of the weight As a color, that which insolubilized a disperse dye, the water-insoluble nature color of the oil color,

direct dye, acid dye, basic dye, etc. by lake-ization, a reactive dye, a metallized dye, etc. are used. These colors have the desirable refining color from which mineral salt was removed.

[0024] As an example of a color C. The I. direct blacks 17, 19, 32, 51, 71, 108, 146, 154, and 166, the C.I. ADDO blacks 2, 7, 24, 26, 31, 52, 63, 112, and 118, the C.I. basic black 2, C.I. direct blues 6, 22, 25, and 71, 90, 106, the C.I. acid blues 9, 22, 40, 59, 93, 102, 104, 113, 117, 120, 167, 229, and 234, the C.I. basic blues 1, 3, 5, 7, 9, 24, 25, 26, 28, and 29, the C.I. direct red 1, 4, and 17, 28, 83, the C.I. acid reds 1, 6, 32, 37, 51, 52, 80, 85, 87, 92, 94, 115, 180, 256, and 315, the 317.C.I. basic red 1, 2, 9, 12, 13, 14, and 37, the C.I. direct yellow 12, 24, and 26, 98, the C.I. acid yellow 11, 17, 23, 25, 29, 42, 61, and 71, the C.I. basic yellow 11, the 28.C.I. direct oranges 34, 39, 44, 46, and 60, the C.I. direct violet 47 and 48, C. I. direct Brown 109, the C.I. direct green 59, the C.I. acid oranges 7 and 19, the C.I. acid violet 49, the C.I. basic violet 7 and 14, and 27 grades can be illustrated. A urea, a dimethyl urea, etc. can also be added as other additives.

[0025] The dispersing element of the pigment detailed—ization—processed about manufacture of record liquid, for example, a dispersant, [whether it distributes in a sand mill, a homogenizer, a ball mill, a paint shaker, an ultrasonic disperser, etc. by mixing water or an aquosity resin, an aquosity solvent, etc., and] Or after kneading beforehand well in 2 roll mills, it distributes further in the above-mentioned sand mill etc., dilution, colloidal silica, a chelating agent, and other additives are mixed with water suitably, and record liquid is manufactured. A high-speed disperser besides stirring by the agitator using the usual feather, an emulsifier, etc. can perform mixed stirring.

[0026] Record liquid is enough filtered with the filter of 3 micrometers or less of apertures before dilution or to the back. It is desirable to filter with a filter 0.45 micrometers or less still more preferably with a filter 1.0 micrometers or less preferably. In advance of filtration of a filter, by centrifugal separation, the thing of a big particle size can also be removed, blinding in filtration with a filter is lessened by this, and the duration of service of a filter becomes long.

[0027] Although record liquid is based also on the method of a recording device, it is desirable to adjust as a liquid of viscosity 0.8 – 15 mPa·s (25 degrees C). 25 – 60 dyn/cm of surface tension is desirable, and the weak alkalinity of pH of 7–9 is desirable.

[0028] Though it is aquosity, since the record liquid manufactured by this invention has remarkably good water resistance, it is suitably used as record liquid for ink jets, and it can also be used for the field of record objects, such as preparation in office, a sign, marking of the corrugated paper used for an out board use since lightfastness is further superior to the color type, numbering, and a bar code.

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EXAMPLE

[Example] Hereafter, this invention is explained based on an example. That it is with the example central part and % shows weight section and weight %, respectively.

Example 1 [the creation method of a salt milling processing pigment]

Crude copper phthalocyanine (Toyo Ink make) 250 section sodium chloride 2500 section diethylene glycol The 200 sections were taught to the 1 gallon kneader of stainless steel, and were kneaded for 3 hours. Next, this mixture was supplied to 2.5l. warm water, and it heated and stirred at about 80 degrees C. After stirring for about 1 hour and considering as the shape of a slurry, filtration and rinsing were repeated 5 times and the watercolor-pigment dispersing element (salt milling processing article) of 50% of solid contents was obtained except for the sodium chloride and the solvent. The following raw material was paid to the sand mill, it distributed for 3 hours, and the concentration record liquid for ink jets was created.

[0030]

[The creation method of dark color record liquid]

Indigo pigment (50% of salt milling processing article solid contents) . Section [30.0 /] Dimethylamino Ethanol The 0.1 sections Dispersant (emulgen 420 by Kao Corp.) The 8.0 sections Purified water The 55.7 sections Glycerol The 6.0 sections Sodium OMAJIN (aurin company make) It mixed with the following after 0.2 partial powder. It filtration and filtered with the 0.45-micrometer membrane filter continuously by the 1-micrometer membrane filter after mixture, and record liquid was manufactured.

[0031]

[The creation method of record liquid]

The above-mentioned concentration record liquid . Section [16.7 /] Colloidal Silica (Snow Tex 30 by Nissan Chemistry Company) The 16.7 sections Dimethylamino ethanol The 0.1 sections Dispersant (emulgen 420 by Kao Corp.) The 1.2 sections Acrylic resin (Japanese polymer company make W-215) The 3.0 sections Sodium OMAJIN (aurin company make) The 0.2 sections Ethylenediaminetetraacetic acid sodium salt The 0.1 sections Glycerol The 10.0 sections Purified water The 52.0 sections. [0032] The filterability when obtaining record liquid was evaluated, viscosity and the mean particle diameter were evaluated as follows about the obtained record liquid, and the clear nature of the injection property when using this record liquid further, a printing state, and a record object, transparency, the water resistance of a record object, etc. were evaluated. A result is shown in Table 1.

The amount currently filtered within [filterable] fixed time with the membrane filter (1 micrometer and 0.45 micrometers) with a diameter of 90mm.

It measured at 25 degrees C using [viscosity] Brookfield viscometer.

It measured with the particle-size-distribution plan ("SALD-1100" by Shimadzu Corp.) of a [mean-particle-diameter] laser diffraction method.

[Printing state] record liquid was put into the cartridge of Epson HG5130, and it recorded on the regular paper (Xerox Corp. make K). Visual evaluation of the printing state of a record object was carried out.

The injection state of the nozzle under [injection property] continuation printing was evaluated with the printing object.

Good: Continuation printing has been correctly carried out in a predetermined position.

Defect: In spite of having carried out continuation printing, a deficit is not produced on the way or it is not printed in a predetermined position.

When it recorded on a [waterproof] regular paper (Xerox Corp. make K), water was hung down to the recording surface or viewing estimated the existence of bleeding of the ink when being under water for 1 minute, and the flow broth of ink after drying what was printed.

Viewing estimated the transparency when carrying out drawdown of the ink to the OHP sheet for [transparency] ink jets, and making it it in a 1.5-mil applicator.

Drawdown was carried out to the [clear nature] regular paper (Xerox Corp. make K), and both C values of Munsell of viewing and a color machine machine (Nippon Denshoku Industries make sigma 80) compared clear nature.

Drawdown was carried out to the [light-fast] regular paper (Xerox Corp. make K), the accelerated test was performed in the fadeometer (ultraviolet-rays long life fadeometer by the SUGA testing-machine company), and both delta E value of viewing and a color machine machine (Nippon Denshoku Industries make sigma 80) compared the grade of the tenebrescence of a color.

[0033] Instead of the crude copper phthalocyanine of example 2 example 1, the Hansa-Yellow system yellow pigment (HOSUTA palm yellow H-3 by Hoechst A.G. G) 260 section was used, and the water dispersing element (salt milling processing article) was obtained like the example 1. Then, the concentration record liquid for ink jets was created like the example 1. The record liquid for ink jets excellent in transparency and clear nature was obtained. An evaluation result is shown in Table 1.

[0034] Instead of the crude copper phthalocyanine of example 3 example 1, the Quinacridone system red-pigments (HOSUTA palm pink E by Hoechst A.G.) 250 section was used, and the water dispersing element (salt milling processing article) was obtained like the example 1. Then, the concentration record liquid for ink jets was created like the example 1. The record liquid for ink jets excellent in transparency and clear nature was obtained. An evaluation result is shown in Table 1.

[0035] Instead of the crude copper phthalocyanine of example 4 example 1, the iso indoline system yellow pigment (PARIO toll yellow D-1819 by BASF A.G.) was used. The concentration record liquid for ink jets was created by the same method as an example 1, with the SORUTOMI link of an example 1 not performed. The record liquid for ink jets excellent in transparency and clear nature was obtained. An evaluation result is shown in Table 1.

[0036] Instead of the crude copper phthalocyanine of example 5 example 1, diamino JIAN truck nil system red pigments (chlromophtal red made from Ciba-Geigy A-2B) were used. The salt milling of an example 1 created the concentration record liquid for ink jets by the same method as an example 1, not carrying out. The record liquid for ink jets excellent in transparency and clear nature was obtained. An evaluation result is shown in Table 1.

[0037] Instead of the crude copper phthalocyanine of example 6 example 1, the indanthrene system blue-pigment (Toyo Ink RIONO gene blue 6505) 250 section was used, and the water dispersing element (salt milling processing article) was obtained like the example 1. Then, the concentration record liquid for ink jets was created like the example 1. The record liquid for ink jets excellent in transparency and clear nature was obtained. An evaluation result is shown in Table 1.

[0038] Without carrying out the same salt milling down stream processing as an example 1 using example 7 carbon black, the following raw material was paid to the sand mill, it distributed for 3 hours, and the concentration record liquid for ink jets was created.

Carbon black (Mitsubishi Chemical # 2600) The 15.0 sections Dimethylamino ethanol The 0.1 sections Dispersant (emulgen 420 by Kao Corp.) The 8.0 sections Purified water The 70.7 sections Glycerol The 6.0 sections Make it be the same as that of an example 1 after that [sodium OMAJIN (product made from aurin) 0.2 section]. The record liquid for ink jets was created. The record liquid for ink jets excellent in transparency and clear nature was obtained.

An evaluation result is shown in Table 1.

[0039] What compensated with the insufficiency by the purified water except for colloidal silica

and the chelating agent at the time of manufacture of record liquid about the pigment used in the one to example of comparison 7 examples 1-7 was used as the concentration record liquid for ink jets of the examples 1-7 of comparison, respectively. Evaluation is performed like an example and a result is shown in Table 2.

the examples 8-11 of comparison — the result which performed evaluation like the example about a certain color type of record liquid (YMCBK) for ink jets is shown in Table 3

[0040] In addition, when the natural dissolution of the record liquid obtained in the examples 1-7 and the record liquid obtained in the examples 1-7 of comparison is carried out after one-week preservation and it is saved by the 60-degree C thermostat at -40 degrees C in January, it holds at -40 degrees C for 3 hours, and applies for 3 hours, and it is to 60 degrees C. When the temperature up was carried out, and it maintenance and cooled to -40 degrees C over 3 hours for 3 hours continuously at 60 degrees C and this was repeated for three days, in the case of which, there is also no generating of settling, early viscosity was maintained, and the injection property was also stable.

[0041]

[Table 1]

	実施例 1	実施例 2	実施例 3	実施例 4	実施例 5	実施例 6	実施例 7
透過性 1 μ 0.45 μ	○ ○						
粘度 cps	3.0	3.5	3.2	2.8	2.5	3.0	2.5
平均粒径 μ m	100	120	110	125	90	100	90
印字状態	◎	◎	◎	◎	◎	◎	◎
噴射特性	○	○	○	○	○	○	○
耐水性	○	○	○	○	○	○	○
透明性	○	○	○	○	○	○	○
鮮明性	○	○	○	○	○	○	○
耐光性	○	○	○	○	○	○	○

[0042]

[Table 2]

	比較例 1	比較例 2	比較例 3	比較例 4	比較例 5	比較例 6	比較例 7
滲過性 1μ 0.45μ	○ ○						
粘度 cps	3.0	3.5	3.2	2.8	2.5	3.0	2.5
平均粒径 μm	100	120	110	125	90	100	90
印字状態	○	○	○	○	○	○	○
噴射特性	○	○	○	○	○	○	○
耐水性	○	○	○	○	○	○	○
透明性	○	○	○	○	○	○	○
鮮明性	×	×	×	×	×	×	×
耐光性	○	○	○	○	○	○	○

[0043]
[Table 3]

	比較例 8 Y	比較例 9 M	比較例11 C	比較例12 BK
透過性 1μ	○	○	○	○
0.45μ	○	○	○	○
粘度 cps	3.1	3.2	3.1	3.0
平均粒径 μm	—	—	—	—
印字狀態	◎	◎	◎	◎
噴射特性	○	○	○	○
耐水性	×	×	×	×
透明性	○	○	○	○
鮮明性	○	○	○	○
耐光性	×	×	×	×

[Translation done.]

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最終頁に続く

(54)【発明の名称】 インクジェット用記録液

(57)【要約】

【課題】一般のオフィス等で使用されている酸性紙、中性紙等のいわゆる普通紙に対し印字記録を行った場合に、記録物の画像品位に悪影響を与えることなく、印字記録物が十分な耐水性および耐光性を有し、且つ長期の保存に対しても問題がなく、更に目詰まりに対しても問題がない高品位な印字を可能にできるインクジェット用記録液を提供する。

【解決手段】顔料、コロイダルシリカ及びキレート剤を水性の液体中に含有してなることを特徴とするインクジェット用記録液。

(2)

【特許請求の範囲】

【請求項1】 顔料、コロイダルシリカ及びキレート剤を水性の液体中に含有してなることを特徴とするインクジェット用記録液。

【請求項2】 顔料を記録液中0.1～10重量%含む請求項1記載のインクジェット用記録液。

【請求項3】 記録液中、コロイダルシリカを0.5～3.0重量%及びキレート剤を0.01～5重量%含む請求項1又は2記載のインクジェット用記録液。

【請求項4】 顔料が、キナクリドン系、フタロシアニン系、アゾ系、イソインドリノン系、ジアンスラキノニルレッド系、スレン系、ペリレン系、カーボンブラックからなる群より選ばれる1種である請求項1記載のインクジェット用記録液。

【請求項5】 更に水性樹脂を含む請求項1～4いずれか記載のインクジェット用記録液。

【発明の詳細な説明】

【0001】

【発明の属する技術分野】本発明は、インクジェット用記録液に関し、一般のオフィス等で使用されている酸性紙、中性紙等のいわゆる普通紙に対する印字記録物の耐水性および耐光性に優れ、且つ高彩度の記録ができる記録液に関する。

【0002】

【従来の技術】従来、インクジェット用記録液としては、酸性染料、直接染料、塩基性染料等の水溶性染料をグリコール系溶剤と水に溶解したものがよく用いられている（例えば、特開昭53-61412、特開昭54-89811、特開昭55-65269）。水溶性染料としては、記録液の安定性を得るために水に対する溶解性の高いものが一般的に用いられる。したがって、インクジェット記録物は一般的に耐水性が悪く、水をこぼしたりすると容易に記録部分の染料のにじみを生じるという問題があった。

【0003】このような耐水性の不良を改良するため、染料の構造を変えたり、塩基性の強い記録液を調製することが試みられている（特開昭56-57862）。また、記録紙と記録液との反応をうまく利用して耐水性の向上を図ることも行われている（特開昭50-49004、特開昭57-36692、特開昭59-20696、特開昭59-146889）。これらの方法は、特定の記録紙については著しい効果をあげているが、記録紙の制約を受けるという点で汎用性に欠け、また特定の記録紙以外を用いた場合には、水溶性染料を使用する記録液では記録物の充分な耐水性が得られないことが多い。

【0004】また、耐水性の良好な記録液としては、油溶性染料を高沸点溶剤に分散しないし溶解したもの、油溶性染料を揮発性の溶剤に溶解したものがあるが、溶剤の臭気や溶剤の排出の問題があり、環境上好ましくない。

2

また、大量の記録を行う場合や装置の設置場所によっては、溶剤回収等が必要になる問題がある。そこで、記録物の耐水性、耐光性をよくするために、水系媒体に顔料を分散した記録液の開発が行われている。インクジェット用の記録液においては、プリンターに高解像度が望まれるにつれノズルの径が細くなってきており、これに伴い着色剤の粒子径も微細化してきている。しかしながら、微細化するとオーバーヘッドプロジェクター等の透明基材への画像形成においては、染料並みの透明性、鮮明性、染料以上の耐光性が可能になるが、記録紙上では染料が記録紙纖維を染色して鮮明な画像が得られるのに対し、顔料分散タイプの記録液（水分散インクジェット記録液）を用いた場合、纖維の間に顔料が落ち込んでしまい下地の記録紙の色を隠蔽することができなくて鮮明性が損なわれる問題が発生している。この問題は顔料の微細化と共に顕著になってきている。鮮明化の向上にはバインダーである水性樹脂の増加により改善はされるが、粘度の著しい増大、吐出安定性に悪影響をきたす。

【0005】

【発明が解決しようとする課題】本発明は上記した従来の方法の問題点を解決し、透明でかつ鮮明な画質が得られる水分散インクジェット用記録液に関する。

【0006】

【課題を解決するための手段】本発明は、顔料、コロイダルシリカ及びキレート剤を水性の液体中に含有してなることを特徴とするインクジェット用記録液に関する。更に本発明は、顔料を記録液中0.1～10重量%含む上記インクジェット用記録液に関する。更に本発明は、記録液中、コロイダルシリカを0.5～3.0重量%及びキレート剤を0.01～5重量%含む上記インクジェット用記録液に関する。更に本発明は、顔料が、キナクリドン系、フタロシアニン系、アゾ系、イソインドリノン系、ジアンスラキノニルレッド系、スレン系、ペリレン系、カーボンブラックからなる群より選ばれる1種である上記インクジェット用記録液に関する。更に本発明は、更に水性樹脂を含む上記インクジェット用記録液に関する。

【0007】

【発明の実施の形態】本発明に用いられる顔料は水に溶解しなければ特に限定されない。不溶性の染料として、分散染料、油溶染料の水不溶性染料、又は直接染料、酸性染料、塩基性染料等をレーキ化により不溶化したものも用いられる。ただし、用いる合成樹脂に完全に溶解する染料は本発明から除外される。

【0008】顔料の例としては、トルイジンレッド、トルイジンマルーン、ハンザエロー、ベンジジンエロー、ピラゾロンレッドなどの不溶性アゾ顔料、リソールレッド、ヘリオボルドー、ピグメントスカーレット、パーマネントレッド2Bなどの溶性アゾ顔料、フタロシアニンブルー、フタロシアニングリーンなどのフタロシアニン

(3)

3

系、キナクリドンレッド、キナクリドンマゼンタなどのキナクリドン系、ペリレンレッド、ペリレンスカーレットなどのペリレン系、イソインドリノンエロー、イソインドリノンオレンジなどのイソインドリノン系、ジアンスラキノニルレッド系、建染染料から誘導されるスレン系、カーボンブラック等がある。

【0009】このような顔料としては、下記の顔料が例示できる。カラーインデックス(C.I.)ナンバーにて示す。C.I.ピグメントエロー12、13、14、17、20、24、74、83、86、93、109、110、117、125、137、138、147、148、153、154、166、161、C.I.ピグメントオレンジ13、16、36、43、51、55、59、61、C.I.ピグメントレッド9、48、49、52、53、57、97、122、123、149、168、177、180、192、215、216、217、220、223、224、226、227、228、238、240、C.I.ピグメントバイオレット19、23、29、30、37、40、50、C.I.ピグメントブルー15、15:1、15:4、15:6、22、60、64、C.I.ピグメントグリーン7、36、C.I.ピグメントブラウン23、25、26、C.I.ピグメントブラック6、7等が例示できる。

【0010】好ましい処理顔料の分散粒子径及び一次粒子径としては、レーザー散乱による測定あるいは電子顕微鏡において平均粒子径が1.0 μm以下、さらに好ましくは、0.2 μm以下である。顔料の分散粒子径及び一次粒子が0.2 μm以上である場合には顔料をソルトミリング処理（無機塩を磨碎助剤とし、少量の溶剤を粘結剤として用いて顔料の一次粒子径を機械的に微細化する処理）を実施してもよい。また、顔料の表面に官能基を付加させた表面処理顔料を用いても良い。このような粒径であると、記録液の製造においての濾過操作が容易であり、記録液の経時での沈降も少なくなる。

【0011】本発明におけるコロイダルシリカは、粒子の表面にシラノール基と水酸基の陰イオンが存在し、無水ケイ酸の超微粒子をコロイド溶液とし、酸化ナトリウムとしてソーダを含有し、安定化させたものであり、コロイドの大きさ（粒径1～100 nm）をもつシリカが水性の液体中に安定に分散しているものである。本発明において用いられるコロイダルシリカは5～100 nmの範囲の大きさの粒子径のものを用いる。好ましくは粒子径が細かく粒度分布の揃った5～60 nmのものである。また粒子径の異なる二種類以上を併用してもよいし、pH調整剤を必要に応じ、適宜調整して、塩基性成分または、酸性成分を加えることもできる。具体的には、スノーテックス（日産化学社製商品名）、ルドックス（デュポン社製商品名）、カタロイド（触媒化学社製商品名）、アデライト（旭電化社製商品名）等が挙げられる。

4

【0012】コロイダルシリカの重量比率は記録液の組成で0.5～30重量%、より好ましくは1～15重量%である。この量が少ないと鮮明性の良好なインクが得られず、一方この量が大きいと保存安定性、吐出安定性の良好なインクが得られない。

【0013】本発明におけるキレート剤は金属イオンと反応してキレート環をつくる。環形成は、共有結合あるいは配位結合またはその双方の生成によって起こる。キレート剤の効果は金属イオンの補足にあるため、記録液中に含有されているコロイダルシリカの表面の金属イオンを補足する。このためコロイダルシリカは準安定状態になり、紙面上で凝集して膜を造り色素の落ち込みが少なくなり、鮮明性が向上する。しかし、キレート剤を含まないと、ヘッドを腐食させないアルカリ領域の記録液とした場合の鮮明性が低下する。

【0014】キレート剤の具体例としては二つの酸性基をもった配位子としては、例えば、マロン酸、シュウ酸、フタル酸、グリコール酸、サリチル酸がある。一つの酸性基と一つの非酸性配位基をもった配位子として、例えば、8-キノリノール、アセチルアセトン、トリフルオロアセトン、ジメチルグリオキシム、ジチゾン、サリチルアルデヒドがある。二つの非酸性配位基をもった配位子としてエチレンジアミン、2,2'-ビピリジン、1,10-フェナントロリンがある。アミノポリカルボン酸としてエチレンジアミン四酢酸、プロピレンジアミン四酢酸、ブチレンジアミン四酢酸、ペンチレンジアミン四酢酸及びこれらのナトリウム塩又はアンモニウム塩等が挙げられる。キレート剤の重量比率はインクジェット用記録液の組成で0.01～5重量%であり、好ましくは0.1～1重量%である。キレート剤の量が下限より少ないと鮮明性の良好なインクが得られず、上限より多いと保存安定性、吐出安定性の良好なインクが得られない。

【0015】顔料をインクジェット用記録液中で沈殿することなく安定に分散させるために分散剤として界面活性剤を用いてもよいが、特に透明性、鮮明性、分散安定性に優れた性能を有するためにはアニオン性あるいはノニオン性の界面活性剤を用いる方が望ましい。界面活性剤としてアニオン性あるいはノニオン性を用いる理由としてコロイダルシリカの表面はマイナスに帯電しており、顔料を安定化するための分散剤がカチオン性だとコロイダルシリカは凝集するためである。アニオン性界面活性剤としては、脂肪酸塩、アルキル硫酸エステル塩、アルキルアリールスルホン酸塩、アルキルナフタレンスルホン酸塩、ジアルキルスルホン酸塩、ジアルキルスルホコハク酸塩、アルキルジアリールエーテルジスルホン酸塩、アルキルリン酸塩、ポリオキシエチレンアルキルエーテル硫酸塩、ポリオキシエチレンアルキルアリールエーテル硫酸塩、ナフタレンスルホン酸フォルマリン縮合物、ポリオキシエチレンアルキルリン酸エステル塩、

(4)

5

グリセロールボレイト脂肪酸エステル、ポリオキシエチレングリセロール脂肪酸エステル等を例示できる。

【0016】ノニオン性界面活性剤としてはポリオキシエチレンアルキルエーテル、ポリオキシエチレンアルキルアリールエーテル、ポリオキシエチレンオキシプロピレンブロックコポリマー、ソルビタン脂肪酸エステル、ポリオキシエキレンソルビタン脂肪酸エステル、ポリオキシエチレンソルビトール脂肪酸エステル、グリセリン脂肪酸エステル、ポリオキシエチレン脂肪酸エステル、ポリオキシエチレンアルキルアミン、フッ素系、シリコン系等を例示できる。

【0017】界面活性剤の使用量には特に限定されないが、一般的にはインクの全重量に対して0.1～1.5重量%の範囲が好ましいが、さらに好ましくは0.1～1.0重量%である。

【0018】本発明のインクジェット用記録液は、記録液の分散安定性や特に印字記録特性を良好とするに、水性の液体のpHが7～10であることが好ましく、特に好ましくは7～9の弱アルカリ性である。pHが6以下であると記録液の安定性が好ましくなく、経時で凝集あるいはゲル化を生じ、10以上ではコロイダルシリカの安定性が悪い。pHの調整にはアミン、無機塩、アンモニア、リン酸、酢酸等を用いることができる。

【0019】本発明のインクジェット用記録液において、水の他に用いられる水性溶剤は、記録液のノズル部分での乾燥、記録液の個化を防止し、安定な記録液の噴射およびノズルの経時での乾燥を防止するものである。このような水性溶剤としてはエチレングリコール、ジエチレングリコール、プロピレングリコール、トリエチレングリコール、トリエチレングリコール、ポリエチレングリコール、グリセリン、テトラエチレングリコール、ジプロピレングリコール、ケトンアルコール、ジエチレングリコールモノブチルエーテル、エチレングリコールモノブチルエーテルエチレングリコールモノエチルエーテル、1、2-ヘキサンジオール、N-メチル-2-ピロリドン、置換ピロリドン、2、4、6-ヘキサントリオール、テトラフルフリルアルコール、4-メトキシベンゼンノン等を例示できる。

【0020】本発明のインクジェット用記録液において、顔料を良好に定着させるために水性樹脂を用いてもよい。この水性樹脂は、水溶解性の樹脂又は分散性の樹脂が用いられる。このような水性樹脂としては、アクリル系、スチレン-アクリル系、ポリエステル系、ポリアミド系、ポリウレタン系等の水溶解性樹脂又は分散性樹脂がある。これらの樹脂は、インクジェット用記録液中に0.5～1.0重量%を含有させることができ、更に好ましくは1～5重量%用いる。0.5重量%よりも少ないと着色剤を十分に定着できない。また、1.0重量%よりも多くなると、記録液の吐出安定性を低下させことがある。なお、水性樹脂として水溶解性の樹

(4)

6

脂を用いた場合、記録液の粘度を高くする傾向があるが、分散性の樹脂では粘度が低く抑えることができたり、また、記録物の耐水性を向上することができる。これらの樹脂は、必要に応じ、アンモニア、アミン、無機アルカリ等の中和剤を適宜調整して加えることができる。

【0021】また、記録液の紙への乾燥を速める目的において見掛けの乾燥性を早くするため浸透剤を加えることができる。このような浸透剤としてメタノール、エタノール、イソプロピルアルコール、ジエチレングリコールモノブチルエーテル等のグリコールエーテル、アルキレングリコール、アルキレンジオール、ポリエチレングリコールモノラウリルエーテル、ラウリル硫酸ナトリウム、ドデシルベンゼンスルホン酸ナトリウム、オレイン酸ナトリウム、ジオクチルスルホン酸ナトリウム等を用いることができる。これらは、記録液の5重量%以下の使用量で十分な効果があり、これよりも多いと印字の滲み、紙抜け（プリントスルー）を起こし好ましくなる。

【0022】本発明の記録液に、黴の発生を防止するために、防黴剤を添加することもできる。具体的には、デヒドロ酢酸ナトリウム、安息香酸ナトリウム、ソジウムピリジンチオナー-1-オキサイド、ジンクピリジンチオナー-1-オキサイド、1, 2-ベンズイソチアゾリン-3-オノン、1-ベンズイソチアゾリン-3-オノンのアミン塩等が用いられる。これらは、記録液の0.05～1.0重量%程度用いることができる。また、記録液の循環、あるいは、移動、また、記録液の製造時の泡の発生を防止するため消泡剤を添加することもできる。

【0023】染料としては、顔料の色相の調整、濃度の付与等を目的として耐水性、耐光性に問題が無いかぎり使用することが可能である。染料の使用によっては、顔料の分散の安定性を悪くすることもあるので、顔料の4.0重量%以下、好ましくは2.5重量%以下の使用に止めが必要がある。染料としては、分散染料、油溶染料の水不溶性染料、直接染料、酸性染料、塩基性染料等をレーキ化により不溶化したもの、反応性染料、含金属染料等が用いられる。これらの染料は、無機塩の除去された精製染料が好ましい。

【0024】染料の具体例としては、C. I. ダイレクトブラック17、19、32、51、71、108、146、154、166、C. I. アッドブラック2、7、24、26、31、52、63、112、118、C. I. ベーシックブラック2、C. I. ダイレクトブルー6、22、25、71、90、106、C. I. アシッドブルー9、22、40、59、93、102、104、113、117、120、167、229、234、C. I. ベイシックブルー1、3、5、7、9、24、25、26、28、29、C. I. ダイレクトレッドド1、4、17、28、83、C. I. アシッドレッド

(5)

7

1、6、32、37、51、52、80、85、87、
 92、94、115、180、256、315、31
 7. C. I. ベーシックレッド1、2、9、12、1
 3、14、37、C. I. ダイレクトエロー12、2
 4、26、98、C. I. アシッドエロー11、17、
 23、25、29、42、61、71、C. I. ベーシ
 ックエロー11、28. C. I. ダイレクトオレンジ3
 4、39、44、46、60、C. I. ダイレクトバイ
 オレット47、48、C. I. ダイレクトブラウン10
 9、C. I. ダイレクトグリーン59、C. I. アシッ
 ドオレンジ7、19、C. I. アシッドバイオレット4
 9、C. I. ベーシックバイオレット7、14、27等
 を例示できる。その他の添加剤として、尿素、ジメチル
 尿素等を加えることもできる。

【0025】記録液の製造については、例えば、微細化処理した顔料の分散体、分散剤、水ないし水性樹脂、水性溶剤等を混合し、サンドミル、ホモジナイザー、ボールミル、ペイントシェーカー、超音波分散機等にて分散するか、あるいは、二本ロールミルにてあらかじめ良く混練したのち、上記サンドミル等にてさらに分散し、適宜水にて希釈、コロイダルシリカ、キレート剤及び他の添加剤を混合して記録液を製造する。混合攪拌は、通常の羽を用いた攪拌機による攪拌のほか、高速の分散機、乳化機等により行うことができる。

【0026】記録液は、希釈の前あるいは、後に孔径3*

[ソルトミリング処理顔料の作成方法]

粗製銅フタロシアニン（東洋インキ社製）
 塩化ナトリウム
 ジエチレングリコール

をステンレス1ガロンニーダーに仕込み、3時間混練した。つぎにこの混合物を2.5リットルの温水に投入し、約80℃に加熱、攪拌した。約1時間攪拌してスラリー状とした後、濾過、水洗を5回繰り返して塩化ナトリウム及び溶剤を除き、固形分50%の水性顔料分散体※

[濃色記録液の作成方法]

藍顔料（ソルトミリング処理品 固形分50%）
 ジメチルアミノエタノール
 分散剤（花王社製エマルゲン420）
 精製水
 グリセリン
 ソジウムオマジン（オーリン社製）

分散後、下記のものと混合した。混合後、1μmのメンブランフィルターにて濾過、続いて0.45μmのメンブランフィルターにて濾過、続いて0.45μmのメンブランフィルターにて濾過し、記録液を製造した。

★【0031】

[記録液の作成方法]

上記濃縮記録液
 コロイダルシリカ（日産化学社製スノーテックス30）
 ジメチルアミノエタノール
 分散剤（花王社製エマルゲン420）
 アクリル樹脂（日本ポリマー社製W-215）
 ソジウムオマジン（オーリン社製）

(5)

8

* μm以下のフィルターにて十分濾過する。好ましくは、1.0μm以下のフィルターにて、さらに好ましくは0.45μm以下のフィルターにて濾過することが好ましい。フィルターの濾過に先立って、遠心分離によつて、大きな粒径のものを除くこともでき、これによつてフィルターによる濾過における目詰まりを少なくし、フィルターの使用期間が長くなる。

【0027】記録液は、記録装置の方式にもよるが、粘度0.8~1.5mPa·s (25℃)の液体として調整することが望ましい。表面張力は、25~60dyn/cmが好ましく、pHは7~9の弱アルカリ性が好ましい。

【0028】本発明により製造される記録液は、水性でありながら耐水性が著しく良好であるのでインクジェット用記録液として好適に用いられ、オフィスにおける書類の作成、記号、更に耐光性が染料タイプより優れていますのでアウトボード用途で用いられるダンボールのマーキング、ナンバリング、バーコード等の記録物の分野に利用することもできる。

【0029】

【実施例】以下、実施例に基づいて本発明を説明する。例中部、%とあるのは、それぞれ重量部、重量%を示す。

実施例1

250部
 2500部
 200部

※（ソルトミリング処理品）を得た。サンドミルに下記の原料を入れ3時間分散し、インクジェット用濃縮記録液を作成した。

【0030】

30.0部
 0.1部
 8.0部
 55.7部
 6.0部
 0.2部

★ブランフィルターにて濾過し、記録液を製造した。

16.7部
 16.7部
 0.1部
 1.2部
 3.0部
 0.2部

(6)

9
 エチレンジアミン四酢酸ナトリウム塩
 グリセリン
 精製水

【0032】記録液を得るときの濾過性を評価し、得られた記録液について下記のようにして粘度、平均粒径を評価し、さらに該記録液を用いた時の噴射特性、印字状態、記録物の鮮明性、透明性および記録物の耐水性等を評価した。結果を表1に示す。

【濾過性】一定時間内に直径90mmの1μmおよび0.45μmのメンブランフィルターにて濾過できた量。

【粘度】B型粘度計を用いて25℃にて測定した。

【平均粒径】レーザー回折方式の粒度分布計（島津製作所社製「S A L D - 1 1 0 0」）で測定した。

【印字状態】記録液をエプソン社製HG5130のカートリッジに入れて普通紙（ゼロックス社製K）に記録を行った。記録物の印字状態を目視評価した。

【噴射特性】連続印字中のノズルの噴射状態を印字物によって評価した。

良：所定位置に正確に連続印字できている。

不良：連続印字したにも関わらず、途中に欠損を生じたり、所定位置に印字されていない。

【耐水性】普通紙（ゼロックス社製K）に記録した場合において、記録面に水を垂らしたり、あるいは印字したものをおろし、1分間水に浸漬した時のインキのにじみの有無、インキの流れだしを目視にて評価した。

【透明性】インクジェット用OHPシートに1.5ミルのアクリル板にてインキを展色した時の透明性を目視にて評価した。

【鮮明性】普通紙（ゼロックス社製K）に展色して鮮明性を目視、カラーマシン機（日本電色工業社製Σ80）のマンセルのC値の両方で比較した。

【耐光性】普通紙（ゼロックス社製K）に展色してフェドメーター（スガ試験機社製紫外線ロングライフフェドメーター）にて促進試験を行い、色の褪色の程度を目視、カラーマシン機（日本電色工業社製Σ80）のΔE値の両方で比較した。

【0033】実施例2

実施例1の粗製銅フタロシアニンの代わりに、ハンザエロー系黄色顔料（ヘキスト社製ホスター・パームエローH-3G）260部を用いて実施例1と同様にして水性分散体（ソルトミリング処理品）を得た。その後、実施例*

カーボンブラック（三菱化学社製#2600）
 ジメチルアミノエタノール
 分散剤（花王社製エマルゲン420）
 精製水
 グリセリン
 ソジウムオマジン（オーリン製）

その後、実施例1と同様にしてインクジェット用記録液を作成した。透明性および鮮明性に優れたインクジェット

10
 0.1部
 10.0部
 52.0部

* 1と同様にしてインクジェット用濃縮記録液を作成した。透明性及び鮮明性に優れたインクジェット用記録液を得た。評価結果を表1に示す。

【0034】実施例3

実施例1の粗製銅フタロシアニンの代わりに、キナクリドン系赤色顔料（ヘキスト社製ホスター・パーム・ピンクE）250部を用いて実施例1と同様にして水性分散体（ソルトミリング処理品）を得た。その後、実施例1と同様にしてインクジェット用濃縮記録液を作成した。透明性及び鮮明性に優れたインクジェット用記録液を得た。評価結果を表1に示す。

【0035】実施例4

実施例1の粗製銅フタロシアニンの代わりに、イソインドリン系黄色顔料（BASF社製パリオトールエローD-1819）を用いた。実施例1のソルトミリングは行わないまま、実施例1と同様の方法でインクジェット用濃縮記録液を作成した。透明性及び鮮明性に優れたインクジェット用記録液を得た。評価結果を表1に示す。

【0036】実施例5

実施例1の粗製銅フタロシアニンの代わりに、ジアミノジアントラニル系赤色顔料（チバガイギー社製クロモフタルレッドA-2B）を用いた。実施例1のソルトミリングは行わないまま、実施例1と同様の方法でインクジェット用濃縮記録液を作成した。透明性及び鮮明性に優れたインクジェット用記録液を得た。評価結果を表1に示す。

【0037】実施例6

実施例1の粗製銅フタロシアニンの代わりに、インダンスレン系青色顔料（東洋インキ社製リオノーゲンブルー6505）250部を用いて実施例1と同様にして水性分散体（ソルトミリング処理品）を得た。その後、実施例1と同様にしてインクジェット用濃縮記録液を作成した。透明性及び鮮明性に優れたインクジェット用記録液を得た。評価結果を表1に示す。

【0038】実施例7

カーボンブラックを用いて、実施例1と同様なソルトミリング処理工程を実施することなく、サンドミルに下記の原料を入れ3時間分散し、インクジェット用濃縮記録液を作成した。

15.0部
 0.1部
 8.0部
 70.7部
 6.0部
 0.2部

ト用記録液を得た。評価結果を表1に示す。

【0039】比較例1～7

(7)

11

実施例1～7で用いた顔料について、記録液の製造時にコロイダルシリカ、キレート剤を除き、その不足分を精製水で補ったものをそれぞれ比較例1～7のインクジェット用濃縮記録液とした。評価は実施例と同様にして行い、結果を表2に示す。

比較例8～11

ある染料タイプのインクジェット用記録液(YMCB K)について、評価を実施例と同様にして行った結果を表3に示す。

【0040】なお、実施例1～7で得られた記録液、比 *10

12

* 比較例1～7で得られた記録液を-40℃にて一週間保存後、自然溶解した場合、60℃の恒温槽にて1月保存した場合、-40℃にて3時間保持し、3時間かけて60℃まで昇温し、60℃にて3時間保持、繰り返して3時間かけて-40℃まで冷却し、これを3日間繰り返した場合、いずれの場合においても、沈澱物の発生も無く、初期の粘度を維持しており、噴射特性も安定していた。

【0041】

【表1】

	実施例 1	実施例 2	実施例 3	実施例 4	実施例 5	実施例 6	実施例 7
濾過性 1 μ	○	○	○	○	○	○	○
0.45 μ	○	○	○	○	○	○	○
粘度 cps	3.0	3.5	3.2	2.8	2.5	3.0	2.5
平均粒径 μ m	100	120	110	125	90	100	90
印字状態	◎	◎	◎	◎	◎	◎	◎
噴射特性	○	○	○	○	○	○	○
耐水性	○	○	○	○	○	○	○
透明性	○	○	○	○	○	○	○
鮮明性	○	○	○	○	○	○	○
耐光性	○	○	○	○	○	○	○

【0042】

【表2】

(8)

13

14

	比較例 1	比較例 2	比較例 3	比較例 4	比較例 5	比較例 6	比較例 7
濾過性 1μ	○	○	○	○	○	○	○
0.45μ	○	○	○	○	○	○	○
粘度 cps	3.0	3.5	3.2	2.8	2.5	3.0	2.5
平均粒径 μm	100	120	110	125	90	100	90
印字状態	○	○	○	○	○	○	○
噴射特性	○	○	○	○	○	○	○
耐水性	○	○	○	○	○	○	○
透明性	○	○	○	○	○	○	○
鮮明性	×	×	×	×	×	×	×
耐光性	○	○	○	○	○	○	○

【0043】

【表3】

(9)

	比較例8 Y	比較例9 M	比較例11 C	比較例12 BK
濾過性 1μ 0.45μ	○ ○	○ ○	○ ○	○ ○
粘度 cps	3.1	3.2	3.1	3.0
平均粒径 μm	—	—	—	—
印字状態	◎	◎	◎	◎
噴射特性	○	○	○	○
耐水性	×	×	×	×
透明性	○	○	○	○
鮮明性	○	○	○	○
耐光性	×	×	×	×

【発明の効果】本発明により、一般のオフィス等で使用されている酸性紙、中性紙等のいわゆる普通紙に対し印字記録を行った場合に、記録物の画像品位に悪影響を与えることなく、印字記録物が十分な耐水性および耐光性を有し、且つ長期の保存に対しても問題がなく、更に目詰まりに対しても問題がない高品位な印字を可能にすることができるインクジェット用記録液を提供することができた。

フロントページの続き

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